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(71)Applicant : MATSUSHITA ELECTRON CORP

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(54) SELECTIVE ETCHING LIQUID

(57)Abstract:

PURPOSE: To evaluate a crystal defect in a silicon film, that is, a stacking fault, a dislocation and a fine defect, without depending on a crystal face.

CONSTITUTION: A selective etching liquid consists of the mixed solution of a nitric acid (HNO₃), an acetic acid (CH₃COOH), water (H₂O) and a hydrofluoric acid (HF). The compositional ratio of the nitric acid to the acetic acid to the water to the hydrofluoric acid is HNO₃ : CH₃COOH : H₂O : HF = X : 3 : 1 : 1 in a capacity factor and the composition of the HNO₃ is set in the extent of 6 to 12 (Provided that when the concentrations of the hydrofluoric, nitric and acetic acids are 50% 61% and 99% respectively).

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CLAIMS

[Claim(s)]

[Claim 1] A nitric acid, an acetic acid, water, and the selection etching reagent that consists of mixed liquor of fluoric acid.

[Claim 2] the presentation ratio of mixed liquor -- a capacity factor -- nitric-acid: -- acetic-acid: -- water: -- the selection etching reagent of the semi-conductor substrate according to claim 1 whose nitric-acid presentation of said nitric acid it is fluoric acid =X:3:1:1 and is within the limits of 6-12.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the selection etching reagent used for evaluating the crystal defect of a semiconductor substrate, for example, silicon, i.e., a stacking fault, and a rearrangement and a minute defect.

[0002]

[Description of the Prior Art] Reduction of the crystal defect of a semi-conductor substrate is called for with quality improvement of a semiconductor device, densification, high integration, and diameter[of macrostomia]-izing of a wafer. The crystal defect of a semi-conductor substrate has a thing accompanying crystal growth, and the thing induction is carried out [a thing] by the process. For example, the thermal stress rearrangement generated by heat treatment, a stacking fault, the secondary defect by the ion implantation, etc. are them. It works as generation of a carrier, and a recombination center, and these crystal defects reduce remarkably the life of the minority carrier in the inside of a device, and cause leakage current. Moreover, a stacking fault and a rearrangement tend to serve as a deposit site of heavy metal or oxygen.

[0003] using conventionally the selection etching reagent which observes the crystal defect of a semi-conductor substrate, for example, a silicon substrate, -- em light work "journal OBU electrochemical society" 1977 The 124th volume It is shown in the 756th page (M. Wright: J. Electrochem. Soc., 124, and 756 (1977)). This selection etching reagent is called light (Wright) liquid, and consists of a powerful oxidizer like chrome oxide (CrO_3), a buffer like water (H_2O) or an acetic acid (CH_3COOH), and fluoric acid (HF).

[0004] This selection etching reagent oxidizes and dissolves this oxide (SiO_2) by HF continuously in a silicon front face with a powerful oxidizer like CrO_3 first. It is because the oxidation rates of the silicon in a defective field and other perfect fields differ that a defect is etched alternatively. Usually, in a defective part, oxidizing velocity is quick, and a defective part serves as a concave and is called an etch pit. Therefore, the crystal defect of a semi-conductor substrate can be evaluated by observing this etch pit.

[0005]

[Problem(s) to be Solved by the Invention] However, in the above-mentioned conventional selection etching reagent, it had the fault of the crystal-face bearing dependency of the defective detection sensitivity by the crystal defect kind having been large, and being hard to evaluate the crystal defect generated in each crystal face with the same selection etching reagent correctly.

[0006] This invention does not solve the above-mentioned conventional trouble, and aims at offering the approach that it cannot call at the crystal face but the crystal defect of silicon, i.e., a stacking fault, a rearrangement, and a minute defect can be evaluated.

[0007]

[Means for Solving the Problem] In order to attain this purpose, it is constituted from a nitric acid, an acetic acid, water, and the mixed liquor of fluoric acid by the selection etching reagent of this invention.

[0008] Moreover, the presentation ratio of said mixed liquor is nitric-acid:acetic-acid:water:fluoric acid = $X:3:1:1$ in a capacity factor, and the presentation of a nitric acid is within the limits of 6-12 (however, when the concentration of 50% and a nitric acid is [the concentration of 61% and an acetic acid] 99% for the concentration of fluoric acid).

[0009]

[Function] By this configuration, the crystal-face bearing dependency of the defective detection sensitivity by the crystal defect kind can be lessened. Moreover, this selection etching reagent oxidizes and dissolves this oxide (SiO_2) by fluoric acid (HF) continuously in a silicon (Si) front face with a powerful oxidizer called a nitric acid (HNO_3) first. Water (H_2O) and an acetic acid (CH_3COOH) work as a buffer, and control the etch rate of silicon here.

[0010] It is because the oxidation rates of the silicon of a defective field and other perfect fields differ that a defect is etched alternatively. Usually, in a defective part, oxidizing velocity is quick, a defective part serves as a concave and an etch pit is formed. Observing this with an optical microscope or a scanning electron microscope can estimate the crystal defect of a semi-conductor substrate.

[0011]

[Example] As a property required of a selection etching reagent, selectivity with that it is easy to control an etch rate, that it is applicable to the crystal face of all semi-conductor substrates, and a crystal defect is large, and is that harmful matter, such as that the crystal defect of every class is detectable and 6 ** Cr, is not included.

[0012] Hereafter, the example of the selection etching reagent of this invention is explained, referring to a drawing. Drawing 1 is drawing showing the HNO_3 capacity-factor dependency of the etch rate of a silicon substrate of this example. Here, the selection etching reagent mixed and produced the reagent of HF (50% of concentration), HNO_3 (61% of concentration), and CH_3COOH (99% of concentration), and H_2O (ultrapure water). A presentation ratio is $\text{HNO}_3(61\%):\text{CH}_3\text{COOH}(99%):\text{H}_2\text{O}:\text{HF}(50\%) = X:3:1:1$, and the presentation X of HNO_3 was changed with 3, 6, 9, 12, and 15. Silicon substrates are P type (100) and 10-15ohms of specific resistance cm. Solution temperature is 25 degrees C.

[0013] If HNO_3 capacity factor is made to increase as shown in drawing 1, the etch rate of a silicon substrate will increase. By changing HNO_3 capacity factor, the etch rate of a selection etching reagent is easily controllable.

[0014] Moreover, HNO_3 capacity factor can obtain an etch rate equivalent to a Wright reagent from drawing 1 in 55 - 70% of range (HNO_3 presentation is the range of 6-12).

[0015] Next, the defective detection sensitivity comparison about the rearrangement on the field (100) of the selection etching reagent and Wright reagent of this invention is shown in drawing 2. Here, the selection etching reagent mixed and produced the reagent of HF (50% of concentration), HNO_3 (61% of concentration), and CH_3COOH (99% of concentration), and H_2O (ultrapure water). A presentation ratio is $\text{HNO}_3(61\%):\text{CH}_3\text{COOH}(99%):\text{H}_2\text{O}:\text{HF}(50\%) = X:3:1:1$, and changed the presentation X of HNO_3 with 6, 9, 12, and 15.

[0016] The defective detection sensitivity comparison about the rearrangement on the field (100) of a Wright reagent and this selection etching reagent was performed. A sample is etched and an etch pit consistency is measured with an optical microscope so that cleavage of the same sample may be carried out to two and the amount of etching may become equal. Although a difference arises in an etch pit consistency since a difference is in the defect density which is latent by the sample, this selection etching reagent has defective detection sensitivity equivalent to a Wright reagent to the rearrangement on a field (100). Moreover, defective detection sensitivity is not dependent on HNO₃ capacity factor.

[0017] Next, the defective detection sensitivity comparison about the bulk stacking fault on the field (110) of this selection etching reagent and a Wright reagent is shown in drawing 3. Here, the selection etching reagent mixed and produced the reagent of HF (50% of concentration), HNO₃ (61% of concentration), and CH₃COOH (99% of concentration), and H₂O (ultrapure water). A presentation ratio is HNO₃(61% of concentration):CH₃COOH(99% of concentration):H₂O:HF(50% of concentration) = X:3:1:1, and changed the presentation X of HNO₃ with 6, 9, 12, and 15.

[0018] The defective detection sensitivity comparison about the bulk stacking fault on the field (110) of a Wright reagent and this selection etching reagent etched the sample, and was performed by measuring an etch pit consistency with an optical microscope so that the amount of etching might become equal about a single sample.

[0019] Drawing 3 shows that this selection etching reagent has the defective detection sensitivity in 55 - 70% of range (HNO₃ presentation is the range of 6-12) with HNO₃ capacity factor higher than a Wright reagent to the bulk stacking fault on a field (110).

[0020] Next, the defective detection sensitivity comparison about the bulk minute defect (BMD) on the field (110) of this selection etching reagent and a Wright reagent is shown in drawing 4. Here, the selection etching reagent mixed and produced the reagent of HF (50% of concentration), HNO₃ (61% of concentration), and CH₃COOH (99% of concentration), and H₂O (ultrapure water). A presentation ratio is HNO₃(61%):CH₃COOH(99%):H₂O:HF(50%) = X:3:1:1, and changed the presentation X of HNO₃ with 6, 9, 12, and 15.

[0021] The defective detection sensitivity comparison about the bulk minute defect (BMD) of a Wright reagent and this selection etching reagent etched the sample, and was performed by measuring an etch pit consistency with an optical microscope so that the amount of etching might become equal about a single sample.

[0022] From drawing 4, this selection etching reagent has the defective detection sensitivity in 55 - 70% of range (HNO₃ presentation is the range of 6-12) with HNO₃ capacity factor higher than a Wright reagent to the minute defect (BMD) on a field (110).

[0023] Moreover, also in the case of the crystal defect on a field (111) etc., this selection etching reagent is applicable.

[0024]

[Effect of the Invention] This invention cannot call at the crystal face, but can evaluate the crystal defect of silicon, i.e., a stacking fault, a rearrangement, and a minute defect. Especially, about evaluation of the upper (110) stacking fault and a minute defect, from the conventional selection etching reagent, defective selectivity is excellent and it is suitable for evaluation of a wafer, and observation of a process induction crystal defect. Moreover, the crystal defect part of a semi-conductor substrate can be identified easily, and detailed observation of a rejected region is enabled. The cause of a defect can be quickly fed back to a semiconductor device production process or a semiconductor device development process, and the effectiveness to yield stability or early development of a semiconductor device can be expected.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] Drawing showing the relation between the nitric-acid (HNO₃) capacity factor in the selection etching reagent of this invention, and the etch rate of a silicon substrate

[Drawing 2] Drawing showing the defective detection sensitivity comparison about the rearrangement of the selection etching reagent and Wright reagent of this invention

[Drawing 3] Drawing showing the defective detection sensitivity comparison about the bulk stacking fault of the selection etching reagent and Wright reagent of this invention

[Drawing 4] Drawing showing the defective detection sensitivity comparison about the bulk minute defect (BMD) of the selection etching reagent and Wright reagent of this invention

[Translation done.]

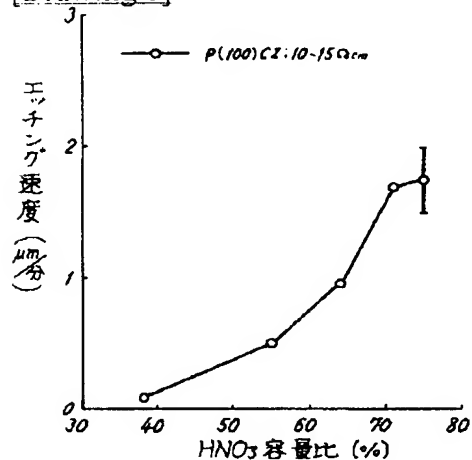
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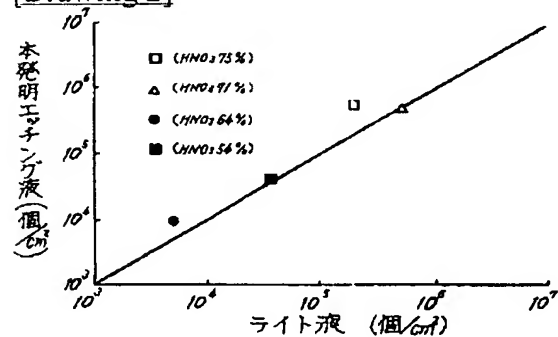
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DRAWINGS

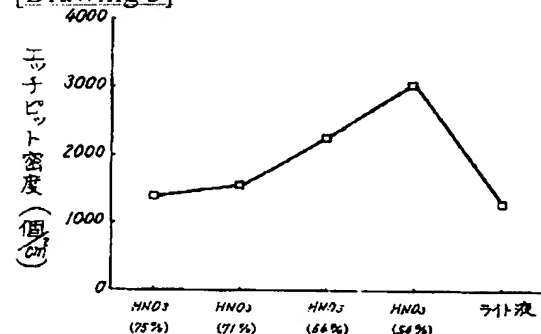
[Drawing 1]



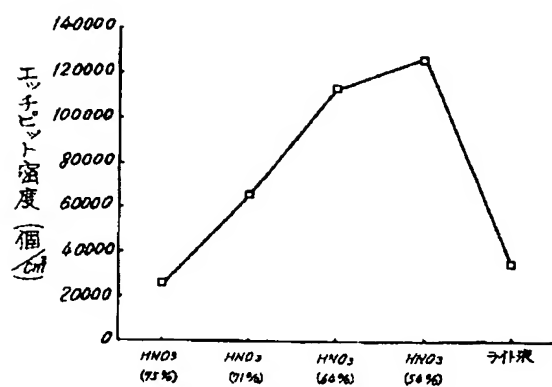
[Drawing 2]



[Drawing 3]



[Drawing 4]



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(54)【発明の名称】 選択エッチング液

(57)【要約】

【目的】 結晶面によらずシリコンの結晶欠陥、すなわち積層欠陥、転位、および微小欠陥を評価する。

【構成】 硝酸(HNO₃):酢酸(CH₃COOH):水(H₂O):弗酸(HF)の混合液からなる。また、組成比として、容量比でHNO₃:CH₃COOH:H₂O:HF=X:3:1:1であり、HNO₃組成が6~12の範囲である(ただし、弗酸の濃度が50%、硝酸の濃度が61%、酢酸の濃度が99%のとき)。

【特許請求の範囲】

【請求項1】 硝酸、酢酸、水、および弗酸の混合液からなる選択エッチング液。

【請求項2】 混合液の組成比が容量比で硝酸：酢酸：水：弗酸=X：3：1：1であり、前記硝酸の硝酸組成が6～12の範囲内である請求項1記載の半導体基板の選択エッチング液。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明は、半導体基板、たとえばシリコンの結晶欠陥、すなわち積層欠陥や転位、微小欠陥を評価するのに使用される選択エッチング液に関するものである。

【0002】

【従来の技術】半導体デバイスの高品質化、高密度化、高集積化、ウェハの大口径化に伴い、半導体基板の結晶欠陥の低減が求められている。半導体基板の結晶欠陥は結晶成長に伴うものやプロセスにより誘起されるものがある。たとえば、熱処理により発生する熱応力転位や積層欠陥、イオン注入による二次欠陥などがそれである。これらの結晶欠陥はキャリアの生成、再結合中心として働き、デバイス中での少数キャリアの寿命をいちじるしく低減させ、リーク電流の原因となる。また、積層欠陥や転位は重金属や酸素の析出サイトとなりやすい。

【0003】従来、半導体基板たとえばシリコン基板の結晶欠陥を観察する選択エッチング液を用いることが、エム・ライト著「ジャーナル オブ エレクトロケミカル ソサエティ」1977年 第124巻 第756ページ (M. Wright: J. Electrochem. Soc., 124, 756, (1977)) に示されている。この選択エッチング液はライト (Wright) 液と呼ばれ、酸化クロム (CrO_3) のような強力な酸化剤と水 (H_2O) や酢酸 (CH_3COOH) のような緩衝剤と弗酸 (HF) とで構成されている。

【0004】この選択エッチング液は、まず CrO_3 のような強力な酸化剤でシリコン表面を酸化し、続いて HF でこの酸化物 (SiO_2) を溶解する。欠陥が選択的にエッチングされるのは、欠陥領域と他の完全領域でのシリコンの酸化速度が異なることによる。通常、欠陥部分では、酸化速度が速く、欠陥部は凹状となり、エッチビットと呼ばれる。したがって、このエッチビットを観察することにより、半導体基板の結晶欠陥を評価することができる。

【0005】

【発明が解決しようとする課題】しかしながら、上記従来の選択エッチング液では結晶欠陥種による欠陥検出感度の結晶面方位依存性が大きく、各結晶面に発生した結晶欠陥を同一選択エッチング液で正確に評価しにくいという欠点を有していた。

【0006】本発明は、上記従来の問題点を解決するもので、結晶面によらずシリコンの結晶欠陥、すなわち積

層欠陥、転位、微小欠陥を評価することができる方法を提供することを目的とする。

【0007】

【課題を解決するための手段】この目的を達成するために本発明の選択エッチング液では、硝酸、酢酸、水、および弗酸の混合液で構成されている。

【0008】また、前記混合液の組成比が容量比で硝酸：酢酸：水：弗酸=X：3：1：1であり、硝酸の組成が6～12の範囲内である（ただし、弗酸の濃度が50%、硝酸の濃度が61%、酢酸の濃度が99%のとき）。

【0009】

【作用】この構成によって、結晶欠陥種による欠陥検出感度の結晶面方位依存性を少なくできる。また、この選択エッチング液は、まず硝酸 (HNO_3) という強力な酸化剤でシリコン (Si) 表面を酸化し、続いて弗酸 (HF) でこの酸化物 (SiO_2) を溶解する。ここで水 (H_2O) や酢酸 (CH_3COOH) は緩衝剤として働き、シリコンのエッチング速度を制御する。

【0010】欠陥が選択的にエッチングされるのは、欠陥領域と他の完全領域とのシリコンの酸化速度が異なることによる。通常、欠陥部分では、酸化速度が速く、欠陥部は凹状となり、エッチビットが形成される。これを光学顕微鏡または走査電子顕微鏡で観察することで、半導体基板の結晶欠陥を評価できる。

【0011】

【実施例】選択エッチング液に要求される特性として、エッチング速度が制御しやすいこと、すべての半導体基板の結晶面に適用できること、結晶欠陥との選択性が大きく、どの種類の結晶欠陥も検出できること、および、6価 Cr 等の有害物質を含まないことである。

【0012】以下、本発明の選択エッチング液の実施例について、図面を参照しながら説明する。図1は本実施例の、シリコン基板のエッチング速度の HNO_3 容量比依存性を示す図である。ここで、選択エッチング液は HF (濃度50%)、 HNO_3 (濃度61%)、 CH_3COOH (濃度99%) の試薬と H_2O (超純水) とを混合して作製した。組成比は HNO_3 (61%) : CH_3COOH (99%) : H_2O : HF (50%) = X : 3 :

1 : 1 であり、 HNO_3 の組成 X は 3, 6, 9, 12, 15 と変化させた。シリコン基板は P 型 (100)、比抵抗 $10 \sim 15 \Omega \text{cm}$ である。液温は 25°C である。

【0013】図1に示すように、 HNO_3 容量比を増加させると、シリコン基板のエッチング速度が増加する。 HNO_3 容量比を変化させることにより、選択エッチング液のエッチング速度を容易に制御することができる。

【0014】また、図1から HNO_3 容量比が 55～70% の範囲 (HNO_3 組成が 6～12 の範囲) では、ライト液と同等のエッチング速度を得ることができる。

【0015】次に、図2に本発明の選択エッチング液と

ライト液の(100)面上の転位に関する欠陥検出感度比較を示す。ここで、選択エッチング液はHF(濃度50%)、HNO₃(濃度61%)、CH₃COOH(濃度99%)の試薬とH₂O(超純水)とを混合して作製した。組成比はHNO₃(61%) : CH₃COOH(99%) : H₂O : HF(50%) = X : 3 : 1 : 1であり、HNO₃の組成Xを6, 9, 12, 15と変化させた。

【0016】ライト液とこの選択エッチング液の(100)面上の転位に関する欠陥検出感度比較を行った。同一試料を二つに劈開して、エッチング量が等しくなるように、試料をエッチングし、光学顕微鏡でエッチビット密度を測定する。試料により潜在する欠陥密度に違いがあるため、エッチビット密度に差が生じるが、この選択エッチング液は(100)面上の転位に対してライト液と同等の欠陥検出感度を持つ。また、欠陥検出感度はHNO₃容量比に依存しない。

【0017】次に、図3にこの選択エッチング液とライト液の(110)面上のバルク積層欠陥に関する欠陥検出感度比較を示す。ここで、選択エッチング液はHF(濃度50%)、HNO₃(濃度61%)、CH₃COOH(濃度99%)の試薬とH₂O(超純水)とを混合して作製した。組成比はHNO₃(濃度61%) : CH₃COOH(濃度99%) : H₂O : HF(濃度50%) = X : 3 : 1 : 1であり、HNO₃の組成Xを6, 9, 12, 15と変化させた。

【0018】ライト液とこの選択エッチング液の(110)面上のバルク積層欠陥に関する欠陥検出感度比較は、単一試料をエッチング量が等しくなるように、試料をエッチングし、光学顕微鏡でエッチビット密度を測定することにより行った。

【0019】図3より、この選択エッチング液は(110)面上のバルク積層欠陥に対してHNO₃容量比が55~70%の範囲(HNO₃組成が6~12の範囲)では、ライト液より高い欠陥検出感度を持つことがわかる。

【0020】次に、図4にこの選択エッチング液とライト液の(110)面上のバルク微小欠陥(BMD)に関する欠陥検出感度比較を示す。ここで、選択エッチング

液はHF(濃度50%)、HNO₃(濃度61%)、CH₃COOH(濃度99%)の試薬とH₂O(超純水)とを混合して作製した。組成比はHNO₃(61%) : CH₃COOH(99%) : H₂O : HF(50%) = X : 3 : 1 : 1であり、HNO₃の組成Xを6, 9, 12, 15と変化させた。

【0021】ライト液とこの選択エッチング液のバルク微小欠陥(BMD)に関する欠陥検出感度比較は、単一試料をエッチング量が等しくなるように、試料をエッチングし、光学顕微鏡でエッチビット密度を測定することで行った。

【0022】図4より、この選択エッチング液は(110)面上の微小欠陥(BMD)に対して、HNO₃容量比が55~70%の範囲(HNO₃組成が6~12の範囲)ではライト液より高い欠陥検出感度を持つ。

【0023】また、この選択エッチング液は(111)面上の結晶欠陥などの場合にも適用できる。

【0024】

【発明の効果】本発明は、結晶面によらずシリコンの結晶欠陥、すなわち積層欠陥、転位、微小欠陥を評価することができる。特に、(110)上の積層欠陥、微小欠陥の評価に関しては、従来の選択エッチング液よりも、欠陥選択性が優れ、ウェハの評価、プロセス誘起結晶欠陥の観察に適している。また、簡単に半導体基板の結晶欠陥箇所が識別でき、欠陥箇所の詳細な観察を可能にする。不良原因を迅速に半導体装置製造工程あるいは半導体装置開発工程へフィードバックでき、半導体装置の歩留まり安定あるいは早期開発への効果が期待できる。

【図面の簡単な説明】

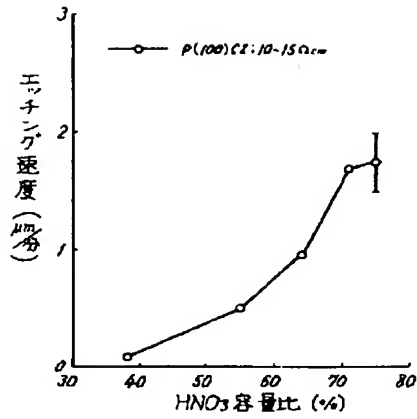
【図1】本発明の選択エッチング液における硝酸(HNO₃)容量比とシリコン基板のエッチング速度との関係を示す図

【図2】本発明の選択エッチング液とライト液の転位に関する欠陥検出感度比較を示す図

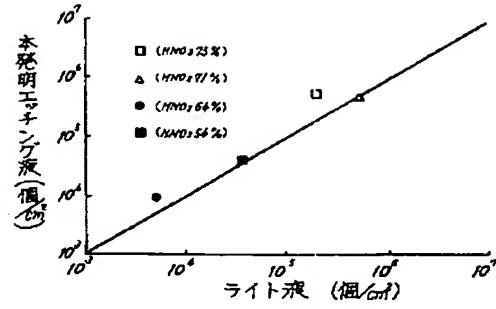
【図3】本発明の選択エッチング液とライト液のバルク積層欠陥に関する欠陥検出感度比較を示す図

【図4】本発明の選択エッチング液とライト液のバルク微小欠陥(BMD)に関する欠陥検出感度比較を示す図

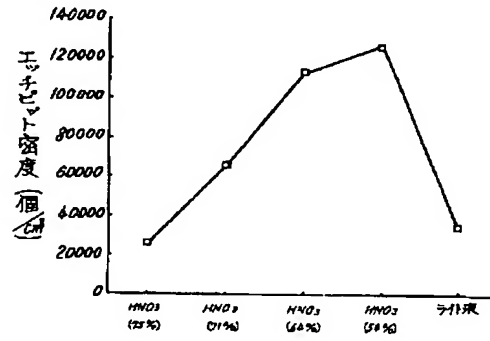
【図1】



【図2】



【図4】



【図3】

